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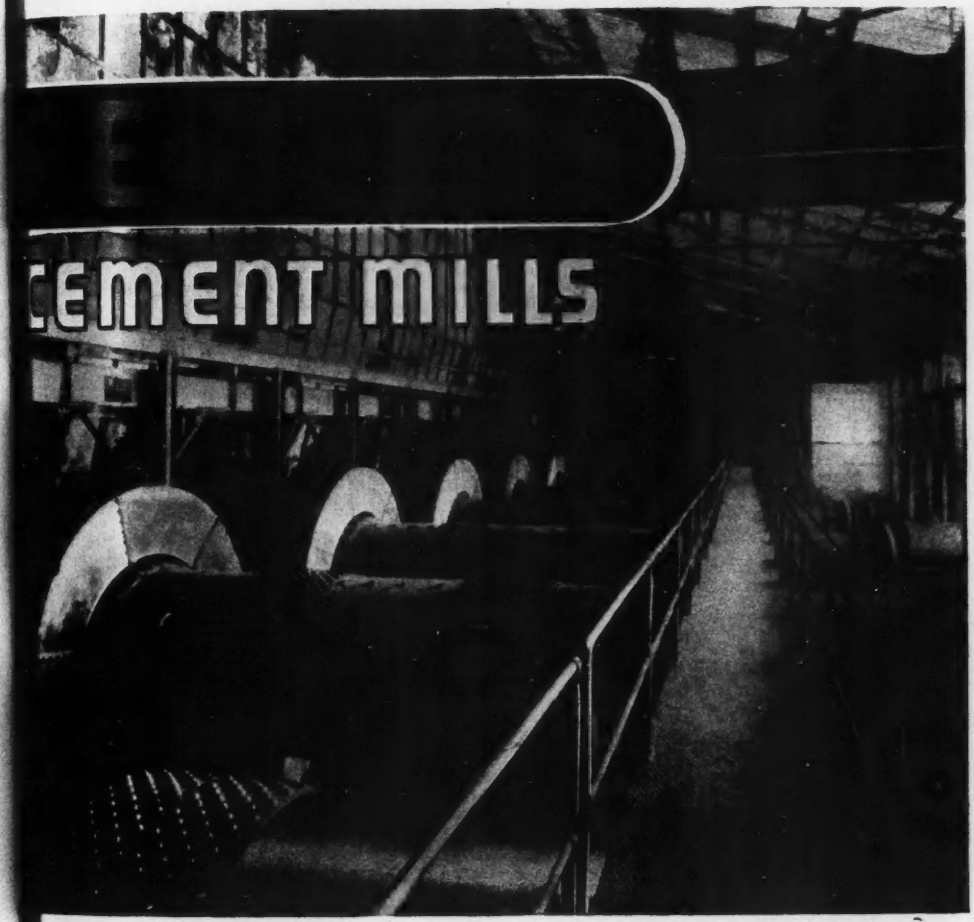

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PUBLISHED BY
CONCRETE PUBLICATIONS LIMITED
14 DARTMOUTH STREET, LONDON, S.W.1

TELEPHONE: WHITEHALL 4581.
TELEGRAPHIC ADDRESS:
CONCRETIUS, PARL, LONDON

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VOLUME XV. NUMBER 7 JULY 1942

Particle Size and Plasticity of Lime.

MEASUREMENTS of the distribution of particle size down to one or two microns and of Emley plasticity values have been made by Mr. D. L. Bishop, and the results of the investigation are published in Research Paper No. RP1232 of the National Bureau of Standards. The particle-size distributions and Emley plasticity values were determined on 25 hydrated limes. The geometric weight-mean diameter varied from 2·9 to 7·8 microns. There was no relation between their particle-size distributions down to 2 microns and their Emley plasticity values. Hydrated limes having very similar particle-size distributions had widely different plasticities. Particle-size measurements down to about 1 micron were made on quicklime putties prepared by hydrating quicklimes with an excess of water. Putties prepared in such a manner were very much finer than the commercial hydrated limes. There was also no relation between the distributions down to 1 micron and the Emley plasticity values of the quicklime putties. It was found that limes having the same calculated specific surface may have widely different particle-size distributions. It is thought possible that the size distribution of the material finer than 2 microns may be the determining factor in the plasticity of limes.

Measurements.

Plasticity measurements of the hydrated limes and quicklime putties were made with the Emley plasticimeter, the instrument commonly used in the lime industry to measure plasticity. The limes were made into stiff putties with water and allowed to soak overnight, after which they were brought to standard consistency by the gradual addition of water accompanied by vigorous stirring, thus obtaining maximum plasticity. The particle-size measurements were made by weighing the lime as it settled out of suspension in a liquid, and calculating the particle-size distribution from the sedimentation record. Anhydrous butanol was used as a dispersion medium. This medium was chosen instead of water because it gives better dispersion of the lime. When lime is dispersed in water, agglomera-

tion takes place, which alters the rate of sedimentation very much and would yield erroneous sedimentation data. In dilute suspensions in water much of the finer fraction is also lost through the interaction of the lime and carbon dioxide, with consequent solution and reduction in size of some of the particles. This seriously affects the particle-size indications, especially of the finer sizes. Sedimentation measurements were therefore made on dry hydrated lime after dispersing it in butanol with the aid of a stirring device.

Particle-Size Distribution.

The sedimentation data of the hydrated limes indicated that many of them contained large amounts of very fine material. This fine material settled so

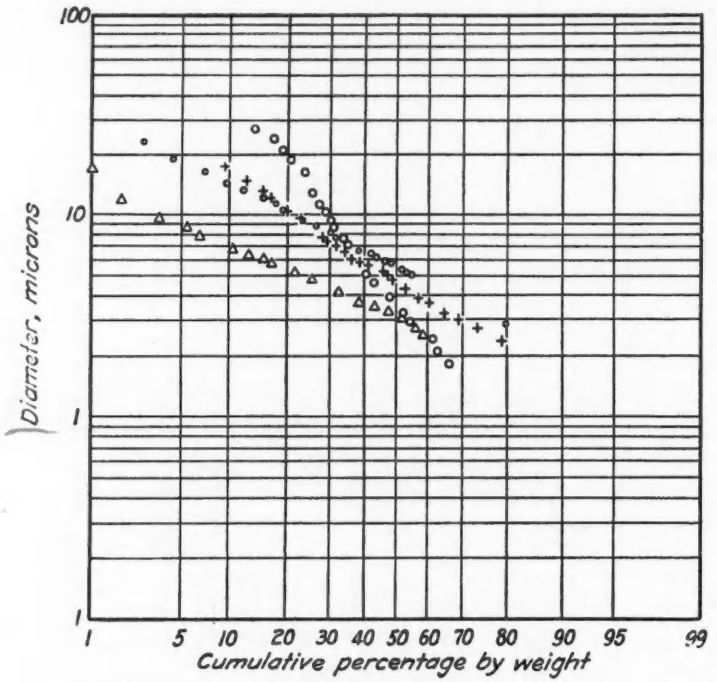

Fig. 1.—Particle-size Distribution of Four Hydrated Limes, showing Straight-line Characteristics.

slowly that it was impractical to obtain a complete sedimentation curve. Hence the sedimentations were ordinarily continued only sufficiently long to give points on the distribution curve down to about 2 microns for the hydrated limes and about 1 micron for quicklime putties.

In order to calculate any of the constants, such as mean diameter or surface area, which are used to describe a distribution, it was necessary to make some assumption regarding the distribution of the material below 2 microns in size.

The cumulative weight distribution of each of the 25 hydrated limes was plotted on logarithmic-probability paper and in each case the points fell very close to a straight line (Fig. 1). It was therefore assumed in obtaining the geometric mean diameters, the geometric standard deviation, and the specific surfaces that for each lime the diameter distribution of the particles smaller than 2 microns was represented by an extrapolation of the straight line fitted to the observed points for particles greater than 2 microns. Hatch and Choate have shown how for such a straight-line distribution the geometric mean and the standard geometric deviation can be obtained graphically, and have pointed out that these two constants completely describe the distribution. For such a distribution the geometric weight-mean diameter, g, is that diameter which is exceeded by particles representing half the total weight of material. On a chart such as Fig. 1 this mean diameter can be read from the ordinate, and corresponds to the position at which a straight line through the plotted points crosses the vertical line representing a cumulative percentage of 50. (The straight lines were fitted by inspection.) The standard geometric deviation, λ, is a measure of the range of the particle sizes and for such a distribution is the ratio of the diameter of the 50 per cent. size to the diameter of the 16 per cent. size.

It can be shown that for such a straight-line distribution the specific surface is given by the formula

$$s = 6 \cdot \frac{\lambda^{\frac{1}{2}\log_e \lambda}}{\rho g}$$

where ρ is the density of the material. (It is assumed that the particles are spherical.) Therefore, the surface per gramme can be computed directly from the values of g and λ, which have been obtained by simple graphical methods. It is of note that for such a distribution any other mean diameter, such as arithmetic-weight mean diameter and surface mean diameter can be quickly calculated from the value of g and λ. The fact that the limes followed very closely the same empirical law of distribution in the entire region in which observations were made is strong evidence that the values reported as geometric mean, geometric standard deviation, and surface per gramme are close to the correct values. However, the surface per gramme depends to such a large extent upon the distribution of the particle diameters below 2 microns that this value is reported only to the nearest 1,000 square centimetres per gramme. One should bear in mind that 50 per cent. by weight of the particles in each lime are of diameters smaller than the values reported as the geometric mean. Since the particle-size distribution was determined for more than 50 per cent. of the weight of every lime, no extrapolation was involved in obtaining the 50 per cent. size. It is only in identifying the 50 per cent. size as the geometric mean that extrapolation is involved. Attention is called to the fact that limes of equal specific surface sometimes had widely different particle-diameter distributions.

Particle Size and Emley Plasticity Values of Hydrates.

There is no relation between the particle-size constants and Emley plasticity values. Some of the nonplastic limes (limes having Emley plasticity values less

than 200) are comparatively coarse, and others are fine. Many of the plastic hydrates are coarse; but it is not likely that this is the determining factor, for the most plastic hydrate is likewise the finest of all. The distribution curves for two plastic and two nonplastic limes, whose particle-size distributions are very similar, are shown in Fig. 2. Not only are all four of these limes very similar, but they may be considered as pairs made of a plastic and a non-plastic lime having almost identical distribution characteristics. Fig. 3 shows the geometric weight-mean diameters, surface per gramme, and Emley plasticity values of the

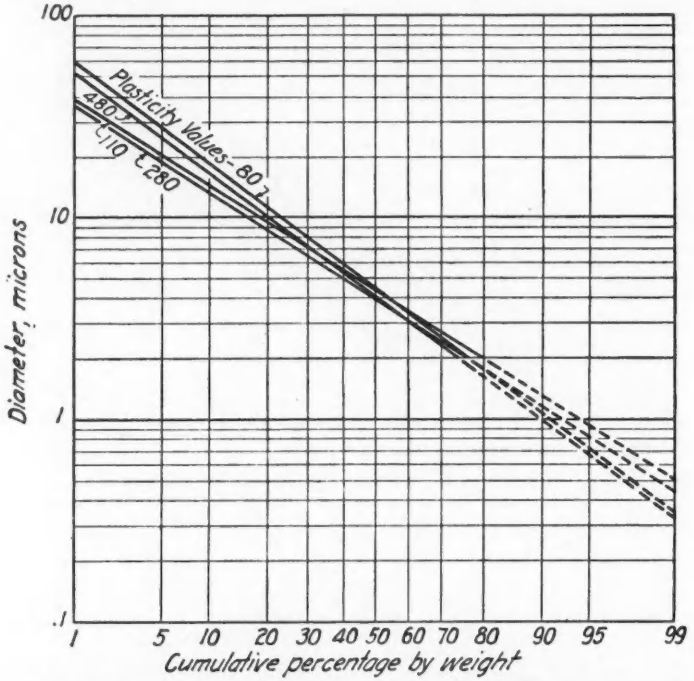

Fig. 2.—Particle-size Distribution of Four Hydrated Limes, having Widely Varying Emley Plasticity Values but Similar Distribution.

hydrated limes. It is apparent that there is no correlation between Emley plasticity values and the geometric means or between such plasticity and specific surfaces. Even though Figs. 2 and 3 do not indicate a relation between particle-size and Emley plastic values, the fact that distributions below 2 microns were not actually measured leaves the possibility that the distribution of the material finer than 2 microns may be the determining factor in plasticity of limes.

Other experiments were made in an effort to find a relation between particle size and Emley plasticity values. An attempt was made to study the plasticity of various sized fractions of limes separated in an air elutriator. There was some increase

in plasticity with decrease in particle size, provided the lime was originally plastic, for separations that could be effected by blowing only a short time in the elutriator. Finer separations, requiring longer time in the elutriator, lost plasticity with increasing time of blowing, either through a slight amount of carbonation or possibly through the loss of an essential fraction of the lime during the elutriation. Such a loss could possibly occur through thermal precipitation in the cooling coils of the elutriator. Separations of non-plastic limes showed no change in Emley plasticity values for the various sized fractions.

A more successful method of making separations of the limes was fractional sedimentation in ethanol. It was found that plastic hydrates could be treated with ethanol without affecting their plasticity. These plastic limes could be washed successively in water, ethanol, butanol, and kerosene, with the result that

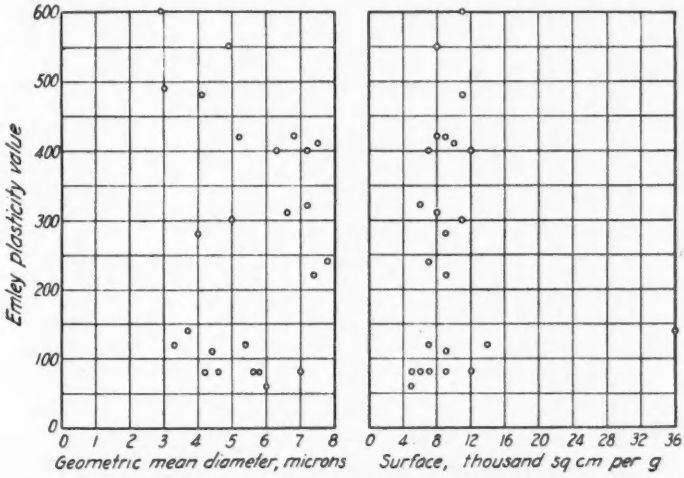

Fig. 3.—Emley Plasticity Values of Hydrated Limes Plotted against their Geometric mean Diameters and Surfaces.

they would be plastic in each liquid. Plasticity measurements on fractions separated in ethanol indicated that for plastic limes the finer fractions of the sample were more plastic than the coarser ones. For non-plastic limes, however, there was no measurable difference in plasticity values for the various fractions.

Particle Size and Emley Plasticity Values of Quicklime Putties.

Particle-size determinations were also made on putties which were obtained by hydrating quicklimes with an excess of water, so as to obtain a wet putty without forming an intermediate dry hydrate. Samples for particle-size determinations were prepared by removing the excess water from the putties with ethanol, and the ethanol in turn with butanol, after which they were dispersed in butanol. Considerable difficulty was encountered in measuring the particle size of these putties with the sedimentation apparatus because of their extreme fineness. Their

sedimentation rates were so slow that even at small depths it was impossible to get complete sedimentation in a reasonable time. The data for these quicklimes are presented as a graph (Fig. 4) because of the uncertainty in calculating distribution constants where such a large percentage of the material finer than 1 micron would have to be calculated from extrapolations. Even though the quicklime putties were very fine, they showed wide variations in particle size. Some contained considerable quantities of comparatively coarse particles, but all had a

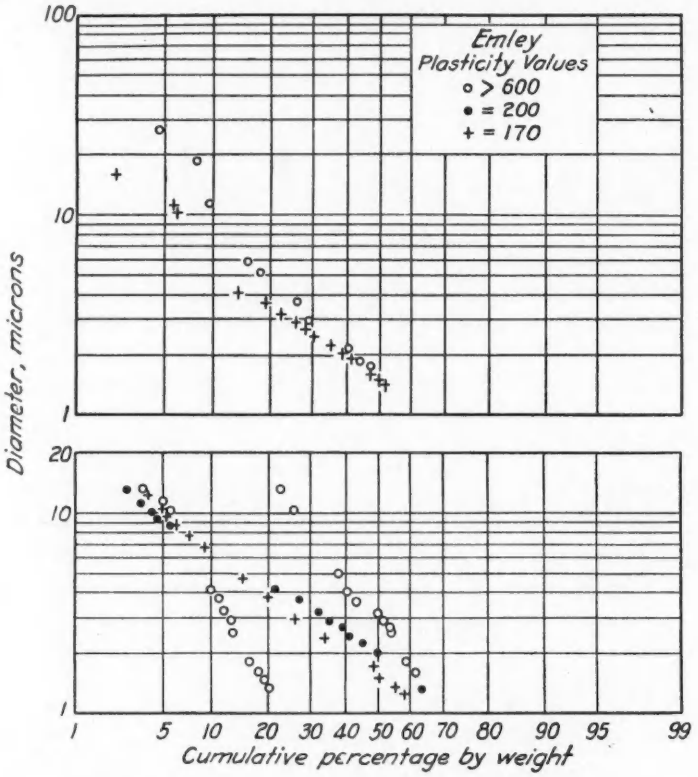

Fig. 4.—Particle-size Distribution of Quick-lime Putties.

large percentage of material finer than 1 micron. Quicklimes, when hydrated with an excess of water, yield products which are very much finer than commercial dry hydrates.

The quicklime putties had Emley plasticity values such that some would be classed as non-plastic and some as very plastic limes. Here again the data (Fig. 4) for the quicklimes indicate that there is no definite relation between Emley plasticity values and particle size, at least down to 1 micron.

Controlling Grinding Mills by Sound.

AN automatic device known as the "Electric Ear" has been developed to record the sound of ball, tube and rod mills and thereby gauge the efficiency of the grinding process, and is stated to be practical and economical. A microphone is mounted in a parabolic reflector to permit it to record only one mill at a time and its energy output is passed through a control circuit, including a relay system.

The character of sound, as well as its intensity, varies when a mill grinding a single material is operated wet or dry, in open or closed circuit, producing a coarse or fine product, and with different feed rates. The blows of balls hitting together or against the lining, with no material between, produce maximum sound. If there is a complete cushion of material between so that energy is dissipated without doing useful work, the balls will be unable to create enough pressure or force to fracture the particles of feed and the sound will be at a minimum. At some point, where there is sufficient material between so that blows of the balls will fracture particles without crushing them unduly, the sound intensity corresponds to the best grinding efficiency. At some best average noise level, this action is more closely approached than at any other.

A change in feed size, other conditions being constant, will cause a change in the noise by altering the spacing between the balls. Since a certain noise level corresponds to optimum grinding within the mill, the feed rate must be changed to match new conditions or the noise level will be different and the mill will operate at less than maximum grinding efficiency. If from feed of the same size and composition a product of different fineness is desired, a different noise level must be established. A higher noise level, other conditions being equal, should correspond to a finer product. This means that the balls are closer together and hit through the individual particles of material, producing a finer product with a greater tendency to overgrind. Conversely, a lower noise level means that the balls are spaced farther apart and their blows are somewhat cushioned. With less crushing action and tendency to overgrind a coarser product is obtained, provided the material flows freely from one end of the mill to the other as in ordinary continuous grinding. When the balls are held apart the volume in the mill is greater, the rate of flow through the mill is faster, and a coarser product is made, since the balls have less opportunity to hit particles. In closed-circuit grinding the rate of passage of material through the mill has an effect on the number of blows the balls can give an individual particle and on the chance of its being underground or overground. Therefore, without varying the noise level or departing from reasonably efficient operation, the fineness may be changed considerably by controlling the circulating load.

The instrument is said to have controlled various types of mills under a wide variety of grinding conditions. In wet grinding it is controlling ball mills from 12in. to 10ft. in diameter, grinding in open and in closed circuit with classifiers. It is also being used with wet grinding rod and tube mills. In dry grinding it is

operating with ball, pebble and tube mills in open circuit and in closed circuit with air classifiers and screens.

It has also been used as an indicator of mill loading without control; the operator observes the sound intensity as registered by the meter and alters the feed or changes the mill control by hand to suit. When grinding wet in open or closed circuit, a change in density materially affects not only the grinding circuit itself but often the subsequent process as well; a method has been devised of using the instrument to maintain the density within the grinding circuit constant.

The instrument can be used to control mill operation automatically only where it is possible to control the feed to the mill by its own operation. In other words, if the feeder is driven by belt, chain or other mechanical means, an electrically operated clutch, ratchet or pawl must be installed to allow the feeder to be actuated directly. Where a mill, such as a regrinding mill, is fed without a regulating feeder and the rate of feed depends on some other operation, the instrument is of no use unless the feeder of the primary mill can be controlled by the sound from the secondary mill. Where two mills are operated in closed circuit with one classifier, and the oversize from this classifier is returned to the two mills, the operation of the instrument is unstable as it is practically impossible to ensure that the oversize material discharged from each mill will be returned to it and not to the other. Several other combinations present similar difficulties.

By controlling the operation of mills by their own sound, operation becomes more stable and the operator is relieved of the necessity of keeping close watch on operating variables. As the mill lining wears down, the change in the noise level is not great enough to require readjustment. If the ball load varies through wear or otherwise, the change is automatically accounted for by sound level. Under normal grinding conditions, whether the grinding is done wet or dry in open or closed circuit to yield a granular or a very fine product, sound bears a definite relation to capacity under a given set of conditions. This sound level will not be the same under all conditions, but there appears to be a sound level which corresponds to optimum results under any given set of conditions. Through the elimination of the variables of manual operation the grinding capacity of a mill is increased, or for the same capacity a finer and more uniform product is obtainable. This statement is based on the results of a good many installations made so far on mills grinding ore, cement, coal, coke, limestone, silica sand, enamel, clay, ilmenite, chrome ore, and other materials. The "Electric Ear" is made by the General Electric Company of the United States.

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Long-Period Tests on Cement.

A VERY extensive series of tests has just been started with a view to studying the effect on concrete of differences in Portland cements. The work is being undertaken by the American Portland Cement Association in accordance with a programme prepared by a committee comprising four representatives of the cement industry and eight research workers and engineers from outside the cement industry.

The objects of the investigation are to learn (1) to what extent differences in cement affect the performance of concrete; and (2) what factors are responsible where differences in performance are found.

A requirement of the committee is that there should be a complete history of each cement during the process of manufacture. In the belief that the best record of the history of a cement during manufacture would be found in the product itself, special emphasis was placed on the collection of samples. Grab samples of the raw materials, the clinker, and the finished cement were taken at frequent intervals, usually every half hour. Where several kilns were used the sampling of each was carried out separately. These grab samples were kept in separate containers so that studies can be made of the uniformity of operations or of the variations in the product. The usual mill control tests, petrographic studies of the clinkers and many special tests were made, or are now in progress, to give the fullest information concerning the cements. One hundred barrels of each of twenty-seven cements and a supply of each clinker are available in sealed drums for use in laboratory studies. As a further record of the uniformity of operation, burning temperatures were taken in the kilns producing the test cement at each plant. The collection of these cements was started in December, 1940, and was completed in June, 1941. More than 24,000 containers were required for the samples.

The second phase of this study, which is now well advanced, is the building of structures of different types and in different localities built in such a manner that, in each series, the different cements will be used under conditions as nearly identical as it is possible to make them under practical conditions.

The first test road was started on July 31 and completed on August 8, 1941. In this road, twenty-four cements were used in sections 75ft. long; each cement was used five times, making a total of 120 sections of 22ft. roadway. During construction observers made moisture determinations and slump tests, checked unit weights and the water content of the mix, made temperature measurements of the slabs, observations on the "bleeding" and finishing characteristics of the cements, and other significant features of the operations. On another test road (finished on November 5) the same twenty-four cements were used; here each cement was used four times, and the sections were 90ft. long. On a test road in New York twenty-seven cements will be used, each four times, in single-line construction; there will also be extra features which will involve the further use

of eighteen of the twenty-seven cements, making in all 180 test sections of single-line road.

At two experimental "farms" all of the test cements are to be used in a number of specimens built in the field and exposed to normal weathering. The specimens are of three types: (1) Slabs 2-ft. 6-in. by 3-ft. 6-in. by 6-in. thick, cast in place; (2) hollow box-like specimens about 2-ft. 6-in. cube in outside dimensions; (3) short columns or posts 8-in. by 8-in. by 60-in. set endwise 2-ft. in the ground. The slab specimens are intended to simulate the exposure in a road slab. The boxes, which are to be kept filled with moist earth, simulate the exposure of concrete members extending from the ground line into the air. For the slab specimens there are three different types of aggregate, each used in three different mixes, making nine rows. For the box and column specimens there are two different types of aggregate, each used in six different mixes. Thus, for each of the twenty-seven cements there are at each farm nine slabs, twelve boxes, and twelve columns. If differences in performance develop which show that certain cements are superior or inferior in resistance, considering all of the structures as a whole, it will be assumed that some property or peculiarity of the cement has affected its concrete-making qualities.

The effect of the exposure of reinforced concrete to sea-water in northern and southern latitudes and to fresh water is to be studied. One hundred and thirty-two concrete piles, 12-in. by 12-in. in cross-section, were made, using twenty-two of the test cements. There were six piles for each cement, three 30-ft. long and three 20-ft. long. The 30-ft. piles were shipped to Buzzards Bay where they were driven by jetting in the Cape Cod Ship Canal. The 20-ft. piles were shipped to New York, where they are available for driving this year in the Hudson River. The concrete was mixed in two different proportions and in two consistencies. Sixty-six similar piles, 30-ft. long, using the same twenty-two cements have been driven in Florida. While the piles for Buzzards Bay were being made, two 6-in. by 6-in. by 30-in. beam specimens were made for each mix and consistency for each cement. These 132 specimens were transported to Maine, where half of them are now exposed to sea water and the other half are in storage for later exposure at the same place. During 1943 an additional group of piles will be exposed to sea-water in southern California. A "farm" will also be established for exposure of specimens to alkali soils.

The test cements are also to be used in thin sections of concrete like parapet walls on two or possibly more structures where they will be exposed to severe changes in weather.

The programme of laboratory tests of clinker and cements requires that every type of laboratory test now recognised or later to be devised which will relate the characteristics of the cement to its performance under service conditions shall be carried out. This part of the work is well under way and data on chemical and physical properties of the cement and clinker are available. Petrographic studies of the clinker are also well advanced. In all the work separate grab samples are being studied as well as composite samples.

New Apparatus for Setting Time Tests.

ILLUSTRATIONS are given in Figs. 1 and 2 of a new apparatus for measuring the setting time of cement, and known as the "Cadograph." It was invented and made by two members of the staff of Southern Portland Cement, Ltd., of Berrima, New South Wales, and is stated by Mr. W. J. Hodge (works manager of the company) to have given excellent results during many months of use. The apparatus is driven by a ¼-h.p. single-phase 240-volt a.c. motor with a speed of 1,450 r.p.m. through a gearbox with a ratio of 15,000 to 1. Six samples in ring moulds may be tested at the same time by placing them on the rotating tables which alternately rotate while the needles are at the highest point of travel.

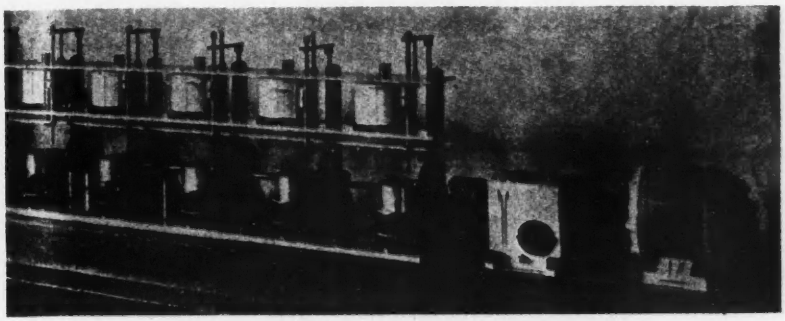


Fig. 1.—Automatic Setting-time Apparatus.

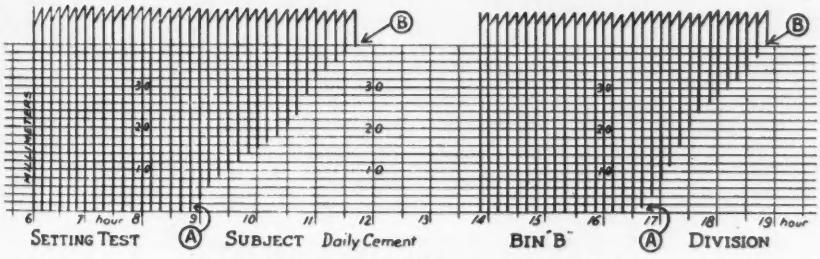


Fig. 2.—Typical Recordings of Setting Time.

The needles, each of which has a cross sectional area of 1 mm. and is weighted to 300 grammes (plus or minus 1 gramme), are lowered into the cement paste at a definite rate of 40 mm. in 60 seconds. Attached to the needle assembly is a pen which records each needle penetration on the chart. When the cement has reached the initial set stage, the pen records 1 mm. from the base line of the chart, as shown at (A) in *Fig.* 2. The final set is reached when the pen records 0·5 mm. from the top line of the chart, as shown at (B). The mechanical movement of the needles, sample tables, and chart drums is controlled by cams mounted on a shaft which revolves at the rate of one revolution in ten minutes, being

driven from the low-speed gear-shaft. The speed reduction from 15,000 to 1 is obtained through three sets of steel worms and phosphor bronze worm wheels fitted into a mild steel case measuring 5 in. by 5 in. by 5 in., the gear ratios being 30 to 1, 25 to 1 and 20 to 1. The gear case is oil-tight, and most of the wheels, worms, and bearings are placed below the normal oil level, the remainder being splash lubricated.

The instrument, with the exception of the gearbox and motor, is fitted into a wood case with glazed top and front, and fitted with thermostatically controlled heaters and water trays to maintain a constant temperature of 70 deg. to 75 deg. F. and a humidity of 96 per cent. The charts are divided vertically into 48 sections, each section representing half an hour and each hour being numbered from 1 to 24. Each horizontal line represents 2 mm. *Fig.* 2 is a typical chart which shows the setting-time curve and rate of setting of two consecutive samples.

Tests on Refractory Mortars.

THE results of a series of tests on the strength of refractory mortars are described by Messrs. R. A. Heindl and W. L. Pendergast in Research Paper RP1461 of the United States National Bureau of Standards. The samples were of the type sold in the dry condition and were investigated in the unheated state with regard to tempering water, fineness of grain, soluble alkali content, pyrometric-cone equivalent, suitability for trowelling and dipping, drying and setting properties, and strength. After heat treatments at various final temperatures the strengths of the mortars were determined. In rupturing assemblages of two half-bricks and mortar, the types of failure were noted. The tendency of the mortars to shrink, crack and flow when exposed to high temperatures, both in fusion blocks and in units of three bricks each, was also investigated. The ingredients of the mortars were all fine-grained, only a small amount being retained on a No. 40 sieve. The strengths of the neat mortars covered a wide range; the strengths of those received and stored in metal drums indicated the desirability of storage in airtight containers. In a series of heat treatments the neat mortars in general were found to have the least strength when preheated at 750 deg. C. However, the least strengths of the majority were noted in assemblages of bricks and mortar preheated at 1,000 deg. C. The mechanical trowelling machine gave a better indication of the workability of the mortars than did the time-of-set test. Observations made on the mortars heated in fusion blocks in general corroborated the shrinkage, cracking and fusion of the same mortars heated in brick piers.

Seventeen air-setting refractory mortars (four of which had a chromite base) marketed in the dry condition and furnished by twelve manufacturers were tested. Different amounts of water were added to the mortars to obtain consistencies suitable for preparing specimens of the neat mortar and for use in bonding bricks by trowelling and dipping methods. Six of the mortars were

furnished in metal drums and the remainder in paper-lined jute sacks. They remained in storage from one week to 49 months before being tested.

The water required for bringing the mortars to a trowelling consistency ranged from 16·6 to 43 per cent., and that required for a dipping consistency ranged from 20·7 to 60·1 per cent. The ease of trowelling on bricks, determined mechanically, was considered satisfactory for all but five of the mortars. The workability of seven of the mortars was considered unsatisfactory when of a dipping consistency. The consistency, as determined with a flow table, ranged from 24 per cent. to 110 per cent., with twelve of the mortars ranging between 62 per cent. and 86 per cent. After slaking in water for one hour, sixteen of the seventeen mortars showed less than 4 per cent. retained on a No. 40 U.S. standard sieve, and in no case was the residue on a No. 20 sieve greater than 0·5 per cent. The free-alkali content, expressed as sodium oxide, ranged from 0 to 1·90 per cent. The pyrometric-cone equivalents of the mortars ranged from 20 to above 38, with very little difference between the end points of calcined and uncalcined materials. Eight of the mortars, mechanically trowelled on bricks and dried, cracked seriously. The time required for the mortars to air-set (indication of water retention), indicated by their resistance to flow under load, ranged from one to thirty minutes when used to bond firebricks of 8·6 per cent. absorption.

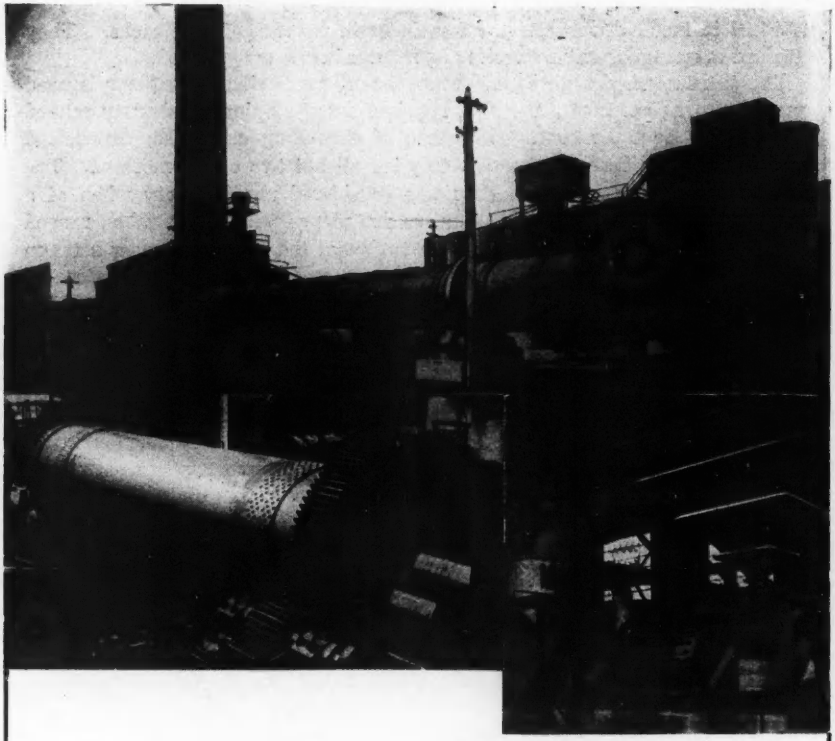
The average transverse dry strengths of the air-set neat mortars ranged from 250 to 4,990 lb. per square inch. With three exceptions, the strength of the test specimens was considerably greater when the surface uppermost during drying was in tension in the breaking test. Eleven of the mortars showed lower strengths after heating at 750 deg. C. than after heating at 1,000 deg. or 1,350 deg. C., four being weakest after air drying and two strongest. The bonding strength of the dried mortars in assemblages of two half-bricks and mortar ranged from 45 to 480 lb. per square inch.

Brick specimens bonded with eleven of the seventeen mortars were weaker after heating at 1,000 deg. C. than after drying or heating at 750 deg. C. Furthermore, seven of the mortars preheated at 1,000 deg. C. had practically no bonding strength. In these tests failure of the assembly occurred either in the bricks, in the mortar, in the joint, or in a combination of these.

Piers of two standard-size bricks and two half-bricks (making two horizontal and one vertical joint) were made with $\frac{1}{8}$in. and $\frac{1}{16}$in. joints. The mortar joints of piers for five of these mortars, all of which had high drying shrinkages, were badly cracked after drying. Also, as a result of the heating at 1,500 deg. C., eight of the mortars had either become fluid enough to bulge from the joints or had cracked. Some of the mortars shipped and stored in sacks, rather than in airtight containers, would undoubtedly have had better strength and bonding properties if they had been tested soon after manufacture.

The failure of a cap of mortar to adhere to brick was found to be no indication of its bonding properties.

A Federal specification for refractory mortar will be based on the results of tests made in this investigation, and will probably cover fineness, bonding strength, refractoriness, and workability.



CEMENT MAKING MACHINERY

The whole resources of this Company being entirely devoted to production for national purposes, it is not possible to quote for delivery abroad until normal conditions return.

VICKERS - ARMSTRONGS LIMITED

Head Office:
VICKERS HOUSE, BROADWAY, LONDON, S.W.1.

Potassium Content of Portland Cement.

What is claimed to be a quick and cheap method for the determination of potassium in Portland cement is described by Mr. W. L. Honaker, of the Ideal Cement Co., in a recent number of our United States contemporary, "Concrete."

The separation of the alkalis from the silicates is effected through evaporation with hydrochloric acid, after which the hydrochloric acid is completely volatilised. The residue is then taken up in dilute acetic acid, and the silica and ferric acetate are eliminated by filtration. Precipitation of the potassium is accomplished at room temperature, using sodium cobalti-nitrite. A centrifuge is used to separate the precipitate, and the precipitate is washed only once. Solution of the potassium sodium cobalti-nitrite is made, using standard ceric sulphate in excess, with the addition of sulphuric acid. The excess ceric sulphate is titrated with standard ferrous ammonium sulphate, using o-phenanthroline-ferrous complex as the indicator.

Reagents.

The reagents are: (1) Hydrochloric acid, 1 : 1. (2) Acetic acid, 1·20. (3) Precipitating reagents: Mix together 46·2 gr. of sodium cobalti-nitrite, 18·9 gr. of sodium acetate, 120 ml. of distilled water, and 18 ml. of glacial acetic acid. Prepare forty-eight hours before using. Keep stoppered, and in a cold, dark place—the cement moist-cabinet is suggested. Before using, centrifuge to remove any precipitate. (4) Ethyl alcohol, 95 and 70 per cent. by volume. (5) Ceric sulphate: Dissolve 9 gr. anhydrous ceric sulphate in 500 ml. of distilled water to which has been added 30 ml. of concentrated sulphuric acid. Make up to one litre. This solution, which is approximately 0·02*N*, may be standardised against ferrous ammonium sulphate. (6) Ferrous ammonium sulphate: Dissolve 8 gr. of ferrous ammonium sulphate ($6\,H_2O$) in 500 ml. of distilled water to which have been added 10 ml. of concentrated sulphuric acid, and make up to one litre. This solution may be standardised against standard potassium dichromate. (7) Sulphuric acid, 1 : 1. (8) Indicator: 0·025*N* o-phenanthroline ferrous complex. Prepare by dissolving 0·49 gr. of ferrous ammonium sulphate in 25 ml. of distilled water, adding 0·743 gr. of o-phenanthroline monohydrate, and stirring until dissolved. Make up to 50 ml. A dark red solution results.

Procedure.

To 2 gr. of Portland cement in a 250-ml. beaker add 25 ml. of 1 : 1 hydrochloric acid while swirling. Break up the residue with a glass rod. Bring the solution to boiling and set it on a water bath, evaporating to complete dryness. Bake it in an oven at from 125 deg. C. to 150 deg. C. for one hour. Cool and take up the residue in 25 ml. of 1 : 20 acetic acid. Bring the solution to boiling and again set it on a water bath, evaporating it with occasional stirring to 15 ml. Add another 10 ml. of 1 : 20 acetic acid and wash down the sides of the beaker until a volume of 40 to 50 ml. is obtained. Again evaporate the solution to 15 ml. on a water bath. Remove and filter through a medium-texture filter paper

into a 100-ml. volumetric flask, washing the residue five or six times with hot water. Cool the filtrate to room temperature and make it up to 100 ml.

To 1·5 ml. of 95 per cent. ethyl alcohol in a 15-ml. centrifuge tube add a 5-ml. aliquot portion of the potassium solution. Mix thoroughly. Add dropwise, with continuous shaking, 2 ml. of the precipitating reagent. Allow it to stand for at least an hour at from 20 deg. C to 25 deg. C. Centrifuge at 2,000 r.p.m. for ten minutes, so that the precipitate is firmly packed at the bottom of the tube. Pour off the supernatant liquid and allow the tube to drain for about five minutes. Wash the precipitate with 5 ml. of 70 per cent. ethyl alcohol, breaking up the bulk of the precipitate with a small glass rod. Centrifuge the solution for five minutes and drain as before. Dry the precipitate for half an hour at from 80 deg. C. to 85 deg. C. to remove all the alcohol. Add 5 ml. of the ceric sulphate reagent and 1 ml. of 1:1 sulphuric acid. Heat in a water bath until the precipitate is completely oxidised as indicated by its disappearance (usually within five minutes). Maintain an excess of ceric sulphate throughout the reaction (5 ml. of 0·02*N* ceric sulphate are sufficient for precipitates containing no more than 0·5 mg. potassium). Transfer the solution to a 125-ml. Erlenmeyer flask, using cold water to wash out the centrifuge tube. Titrate with 0·02*N* ferrous ammonium sulphate, using one drop of o-phenanthroline-ferrous complex as the indicator. The end point is very sharp, the colour of the solution changing from a pale blue to a red.

The calculations are: Ml. of ceric sulphate used in the oxidation of the precipitate, times its normality, times the empirical factor (6·52) equals mg. of potassium in the aliquot portion. Mg. potassium as derived on the above basis, times 1,000, times 1·2046, equals per cent. K_2O.

The most consistent results are obtained on aliquot portions ranging in potassium content from 0·2 mg. to 1 mg. per 5 ml. aliquot portion. Aliquots larger than 5 ml. are not satisfactory. Exact recovery of potassium from solutions containing more than 1 mg. per 5 ml. aliquot portion is questionable. For amounts of potassium in excess of 0·2 mg. per aliquot portion ordinary room temperatures are satisfactory. Low temperatures tend to give high results and high temperatures produce low results, due to difference in the solubility and composition of the precipitate. One washing of the precipitate with 5 ml. of 70 per cent. ethyl alcohol is sufficient. This results in a considerable saving of time. It has been shown that sodium, calcium, magnesium, barium, strontium, zinc, iron, sulphate, chloride, nitrate, and phosphate ions do not interfere in this procedure. However, the precipitate should be kept away from ammonia fumes, and steps should be taken to eliminate ammonia in the reagents and in the sample.

The factor, 6·52, used in calculating the precipitate to potassium is empirical; the results of Drushel and others have established this factor. It is assumed that the precipitate varies between $K_2NaCo(NO_2)_6$ and $KNa_2Co(NO_2)_6$, and that 6·52 is the stoichiometric factor for an intermediate compound. The procedure is of definite importance when applied to cement analysis, both from the standpoints of speed and expense.